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Microwave Conditioning of Durum Wheat. 2. Optimization of Semolina Yield and Spaghetti Quality

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Crosby durum wheat was conditioned with microwave energy at 2450 MHz before experimental macromilling to increase semolina yield and spaghetti quality. Randomly selected duplicate samples were irradiated at 625 W up to 90 s, in 15-s increments. Conditioning parameters were selected on the basis of quality data and grain temperatures observed during application of wide ranges of microwave power (Doty and Baker, 1977) and the results of a micromilling study. In the micromilling study, microwave conditioning was performed at 390 W in 2-s increments up to a maximum grain temperature of 65 °C. Semolina and spaghetti quality parameters of the micromilled samples were not influenced by conditioning time but selectively influenced by level of tempered moisture. Microwave conditioning for 60 s was optimum in the macromilling study. Semolina and semolina plus flour yield was increased 1.8 and 2.5 percentage points, respectively. Spaghetti color, cooked weight, and cooking loss were not altered by this conditioning treatment, while spaghetti firmness was increased significantly.

Procedures for conditioning wheat have been reviewed (Bradbury et al., 1960). Conditioning, the process of adding or removing water and/or heat, creates a moisture gradient within the kernel, which improves milling behavior and quality characteristics of intermediate and finished products. The time required for this internal moisture distribution is shortened when above ambient temperatures are used (Bradbury et al., 1960). The effects of conditioning temperatures above 46 °C have been studied (Geddes, 1929; Becker and Sallans, 1956). Durum wheat has been conditioned at 60 °C without adverse effects on milling and product quality (Seyam et al., 1973).

The use of microwave energy as a heat source would produce highly efficient conditioning, as water molecules distributed throughout the kernel reinforce the three-dimensional distribution of microwave-generated heat (Tape, 1970). The destruction of biologically active molecules following microwave irradiation is due to thermal effects (Goldblith et al., 1968), especially with molecules whose structural integrities result from extensive hydrogen bonding (Takashima, 1962). The biochemical functionings of various molecules are not destroyed under

appropriate irradiation conditions (Goldblith et al., 1968; Takashima, 1966; Ward et al., 1975).

The present study was undertaken to optimize semolina yield and spaghetti quality from durum wheat conditioned with controlled, short periods of microwave power. The fact that short periods of microwave power does not lower semolina and spaghetti quality (Doty and Baker, 1977) suggested the possibility of quality optimization. The results of a micromilling study of microwave conditioned durum wheat, presented herein, suggested the potential of yield optimization.

MATERIALS AND METHODS

Sample Preparation. The sample used in this study was prepared in the same manner previously described (Doty and Baker, 1977).

Conditioning and Milling. Cleaned samples to be micromilled were tempered to 14.5% (w/w) moisture 48 h prior to a second tempering in sealed glass containers to a final moisture of 15.0, 15.5, or 16.0% (w/w) for 24 h. Randomly selected samples were irradiated in 2-s increments, up to a maximum grain temperature of 65 °C, 2 h before micromilling. Total sample weight was 200 g.

Cleaned samples to be macromilled were tempered to 12.5% (w/w) moisture 48 h prior to a second tempering to a final moisture of 17.0% (w/w) 24 h before milling. Randomly selected, duplicate sample (replicates) were sealed in polyethylene containers and irradiated up to 90

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Table I. Range (R), Mean (M), and Standard Deviation (S) of Milling, Semolina Quality, Processing, and Spaghetti Quality Data of 200-g Crosby Durum Wheat Samples after Microwave Conditioning at Different Tempered Moisture Contents

	Tempered moisture								
	15.0% ^a			15.5% ^b			16.0% ^c		
	R	M	S	R	M	S	R	M	S
Milling									
Extraction rate, ^d %	50.1-55.3	53.1	1.4	52.3-56.4	53.5	1.3	51.3-58.9	53.9	2.0
Ash, ^d %	0.700-0.830	0.752	0.029	0.718-0.782	0.748	0.020	0.729-0.834	0.758	0.027
Protein, ^d %	12.2-12.6	12.4	0.2	12.3-13.5	12.6	0.3	12.4-12.7	12.6	0.1
Moisture, %	13.7-14.0	13.8	0.1	14.2-14.5	14.4	0.1	14.0-14.8	14.6	0.2
Semolina quality									
Color score ^e	11.0-11.5	11.5	0.1	11.0-11.5	11.3	0.3	11.0-11.5	11.2	0.3
Specks, no./64.5 cm ²	10-43	28	8	17-47	31	10	23-47	35	10
Starch damage, A ₅₅₀	0.340-0.390	0.371	0.025	0.310-0.425	0.360	0.030	0.250-0.375	0.327	0.039
Lipoxygenase, ΔA ₂₃₄ min ⁻¹ mL ⁻¹	3.2-10.9	6.4	1.7	3.6-8.2	5.9	1.4	3.4-7.8	5.1	1.4
Processing									
Semolina absorption, %	32.3-32.7	32.5	0.2	32.3-33.0	32.4	0.2	32.3-35.0	33.2	0.8
Pressure, lb/in. ²	490-525	510	11	500-540	525	11	415-530	496	11
Spaghetti quality									
Color score ^f	7.5-8.0	7.7	0.2	7.5-8.0	7.8	0.2	8.0-8.5	8.2	0.2
Cooked weight, g	36.5-39.8	38.0	0.9	36.9-39.9	38.1	0.9	36.3-39.8	37.9	1.1
Cooking loss, %	6.3-7.3	6.8	0.3	6.3-7.5	6.8	0.4	6.1-7.6	6.9	0.4
Firmness score, g-cm	4.04-4.72	4.38	0.22	3.94-4.62	4.31	0.21	3.82-4.60	4.24	0.22

^a Thirteen observations, 0 to 26 s of conditioning (2 s data excluded, see text). ^b Thirteen observations, 0 to 24 s of conditioning. ^c Twelve observations, 0 to 22 s of conditioning. ^d 14% moisture basis. ^e Ranges 1.0 (poorest) to 14.0 (best). ^f Ranges 1.0 (poorest) to 11.0 (best).

s, 15-s increments, immediately before milling. Sample temperature was recorded during the time the sample was in the mill hopper. Total sample weight was 2000 g.

Experimentally conditioned samples were micromilled on a Brabender Quadrumat Jr. Laboratory mill (C. W. Brabender Instruments Inc., South Hackensack, N.J.) specifically designed for milling durum wheat. Semolina was recovered by passing the milled stock through a laboratory micropurifier (Vasiljevic et al., 1977). Extraction rates were calculated on total product basis.

Experimentally conditioned samples were macromilled on a Buhler automatic laboratory mill (Buhler-Miag, Minneapolis, Minn.), according to the procedure of Black and Bushuk (1967). Semolina blending and calculation of extraction rates have been described (Doty and Baker, 1977).

Microwave energy was generated as previously described (Doty and Baker, 1977). Samples for micromilling were irradiated at 390 W, while those for macromilling were treated at 625 W.

Physical and Chemical Tests. The procedures used for each physical and chemical test were identical with those of the previous manuscript (Doty and Baker, 1977). Each macromilled replicate was analyzed in duplicate.

RESULTS AND DISCUSSION

Micromilled Samples. The range, mean, and standard deviation of the milling, semolina quality, processing, and spaghetti quality data of the 200-g samples after microwave conditioning are presented in Table I. As expected (Gorakhpurwalla et al., 1975), the time required to reach the final temperature of 65 °C decreased as moisture level increased (26 s at 15.0%, 24 s at 15.5%, and 22 s at 16.0%). The 15.0% moisture sample conditioned for 2 s was not milled and analyzed because of no measurable change in sample temperature after this treatment.

The means of Table I were examined by t-distribution to determine if the observed differences were significant. The results are summarized in Table II. The significant

Table II. Summary of Significant Differences by t-Distribution Analysis of Mean Milling, Semolina, Processing, and Spaghetti Quality Parameters of Table I

	Tempered moisture pair ^a		
	15.0-15.5%	15.5-16.0%	15.0-16.0%
Moisture	1	1	1
Semolina color score	5		5
Starch damage		5	1
Lipoxygenase			5
Semolina absorption		1	1
Processing pressure	1	1	
Spaghetti color score		1	1

^a 1 = 1% level of significance; 5 = 5% level of significance.

increase in semolina moisture probably reflected the increasing tempered moisture levels. Semolina color scores of the 15.0% moisture samples were, on the average, significantly higher than those of the other two moisture levels, while average starch damage of the 16.0% moisture samples was significantly lower. Average semolina absorption and spaghetti color of the 16.0% moisture samples were significantly higher than those of the lower tempered moisture levels. Processing pressure, on the average, of the 15.5% moisture samples was significantly higher than that of the 15.0% and 16.0% moisture samples. Samples tempered to 16.0% moisture are preferred because lower starch damage, lipoxygenase activity, and processing pressure were associated with high semolina and spaghetti color after microwave treatment.

Conditioning time and level of tempered moisture were used as independent, causal variables in an analysis of variance (ANOVA) to further study the interactive effects of microwave conditioning and moisture on quality. The results of the ANOVA, using conditioning time and tempered moisture as the source of variation and within sample variation, respectively, are shown in Table III.

Table III. Effect of Microwave Conditioning Time and Level of Tempered Moisture on Semolina and Spaghetti Quality

Quality parameter	F_{calcd}	
	Conditioning time ^a	Tempered moisture ^b
Milling		
Extraction rate	0.91	0.90
Ash	1.41	0.56
Protein	1.32	1.41
Moisture	0.21	76.15
Semolina quality		
Color score	1.21	3.42
Specks	0.20	1.71
Starch damage	0.33	6.62
Lipoxygenase activity	0.37	2.32
Processing		
Semolina absorption	0.48	11.99
Spaghetti processing pressure	1.33	6.99
Spaghetti quality		
Color score	0.48	11.93
Cooked weight	0.90	0.08
Cooking loss	1.52	0.14
Firmness score	0.46	1.24

^a $F_{0.05} = 2.15$; $F_{0.01} = 2.98$. ^b $F_{0.05} = 3.27$; $F_{0.01} = 5.27$.

Time of microwave conditioning did not significantly influence quality. The range of microwave power used did not reduce semolina or spaghetti quality. On the other hand, semolina moisture, semolina and spaghetti color, starch damage, semolina absorption, and processing pressure were significantly influenced by tempered moisture level during microwave treatment. All were significant at the 1% level except semolina color, which was significant at the 5% level. This influence of moisture on starch damage during microwave conditioning reinforces the requirement for further study of this interaction (Doty and Baker, 1977).

Analysis of the raw data indicated that a random, nonpredictable scattering was operative in all cases except semolina extraction. The variability of each quality factor, except extraction, was not foretellable from one set of conditioning times and tempered moisture level to others. However, extraction was increased after 2 and 12, 6 and 18, and 10 and 24 s of microwave conditioning at 16.0, 15.5, and 15.0% moisture, respectively. The differences of 4 and 6 s between the points of extraction increases at the three moisture levels are readily discernible. The higher the moisture, the lower the time of microwave treatment, thus energy requirements, needed for increased semolina yield.

Optimal Microwave Conditioning. The multimodal response of semolina yield to tempered moisture and time of microwave conditioning nullified the use of linear optimization techniques and complicated nonlinear optimization approaches. The selection of the correct combination of conditioning time and tempered moisture for increasing semolina yield and product quality was based on inspection of all data.

Microwave conditioning times of 0 to 90 s at 625 W were selected because semolina and spaghetti quality is not reduced and grain temperatures do not exceed 40 °C in this span (Doty and Baker, 1977). Attempts to optimize semolina yield were confined to samples of at least 16.0% moisture. At this moisture level, starch damage, lipoxygenase activity, and processing pressure are lower than at lower moisture levels (Table I). Semolina color observed at 16.0% moisture is identical with that at 15.0% for all practical purposes. In addition, maximum spaghetti color occurred at 16.0% moisture. Speckiness and overall

Table IV. Macromilling Data of Semolina Derived from Crosby Durum Wheat after Microwave Conditioning^a

Micro-wave time, s	Grain temperature, °C	Semolina extraction, ^b %	Flour extraction, ^b %	Semolina + flour extraction, ^b %
0	23	56.7	7.1	63.8
15	27	56.3	7.6	63.8
30	28	56.3	7.6	63.9
45	32	56.0	7.4	63.4
60	34	58.5	7.8	66.3
75	38	56.3	7.7	64.0
90	40	56.7	8.3	65.0

^a Each entry is the average of two observations. ^b 14% moisture basis.

Table V. Quality Data of Spaghetti Derived from Crosby Durum Wheat after Microwave Conditioning^a

Micro-wave time, s	Color score ^b	Cooked weight, g	Cooking loss, %	Firmness score, g-cm
0	9.5	38.7	6.0	4.39
60	9.5	39.1	5.8	4.95

^a Each entry is the average of four observations. ^b Ranges 1.0 (poorest) to 10.5 (best).

cooking quality are not reduced at this moisture. Moreover, only 0.19 kcal (2 s at 390 W) and 1.12 kcal (12 s at 390 W) of microwave energy were required to increase semolina extraction 2.7 and 5.8 percentage points, respectively. These minimal energy inputs did not reverse other quality factors (Table III). This increased rate of extraction would be observed in macromilled samples (Vasiljevic et al., 1977).

The macromilling data of the replicated, microwave conditioned samples are shown in Table IV. The semolina physical and chemical data are not reported because they were not significantly different from those of the previous study (Doty and Baker, 1977). After 60 s of microwave conditioning, the rate of semolina and semolina plus flour extraction increased 1.8 and 2.5 percentage points, respectively. The yield of durum flour was 17% higher after the 90 s of microwave conditioning. The semolina recovered after 60 s of microwave treatment was characterized by good color and low speckiness.

These samples were tempered to 17.0% moisture for 24 h prior to microwave conditioning and milling. This procedure differs from the 40-min final temper used in the wide power range study (Doty and Baker, 1977). The reason for the 24-h final temper was to create a uniform, or nearly so, distribution of moisture within the kernel. Application of microwave energy to this system produced optimal increases in semolina extraction, compared with the 40-min final temper, which averaged 0.7 to 0.9 percentage points. It appears that the applied microwave energy recreated a moisture gradient within the kernel before milling. The nature of the type of conditioning achieved by microwave treatment can be detailed only after further *in vivo* studies.

Each 0- and 60-s microwave conditioned semolina sample was processed into duplicate spaghetti samples at an absorption of 34.0% and 465 lb/in.² pressure (Doty and Baker, 1977). The spaghetti quality data of these samples are listed in Table V. A one-way ANOVA of these data showed there was no significant effect on spaghetti cooked weight and cooking loss, after 60 s of microwave conditioning. However, there is a highly significant increase in spaghetti firmness. These trends were not expected on the basis of the gluten and starch physicochemistry observed after 120 s of microwave conditioning (Doty and Baker,

1977). This significant increase reinforces the requirements for further studies on the interaction of microwave energy and gluten and starch before definite conclusions are possible. Although the increase in firmness is significant, the magnitude of the change should not be discernible. The increased firmness increases the cooking quality of the spaghetti. As expected (Doty and Baker, 1977), spaghetti color was not reduced by 60 s of microwave conditioning.

In summary, on the basis of 200-g sample data, microwave conditioning should be confined to samples of at least 16.0% tempered moisture. At this moisture level, samples are characterized by decreased starch damage, lipoxygenase activity, and processing pressure. Semolina color and speckiness are not reduced, while spaghetti color is maximum, the higher the moisture level. Overall spaghetti cooking quality is not decreased by microwave conditioning durum wheat at 16.0% moisture. In addition, minimal energy requirements of 0.19 and 1.12 kcal increase semolina extraction 2.7 and 5.8 percentage points, respectively. Application of 8.96 kcal (60 s at 625 W) of microwave energy to 2000-g samples increases semolina extraction and spaghetti firmness. Improvement of product yield and quality justifies the minimal alteration in tempering required for optimal results. In addition, the energy expenditures are minimal. The feasibility of conditioning durum wheat with microwave energy (Watkins, 1971) is confirmed.

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Acidic Butanol Removal of Color-Forming Phenols from Sunflower Meal

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This paper describes the ability of acidic 1-butanol to remove color-forming phenols (chlorogenic and caffeic acid) and oligosaccharides from sunflower meal without detectable protein denaturation. Defatted sunflower meal of Ala variety was repeatedly extracted with a solution of 1-butanol and 0.005 N HCl (92:8, v/v), giving a protein concentrate (70% protein) with a low chlorogenic residual content ($\leq 0.05\%$). Protein extraction of this product at pH 9.5 and subsequent precipitation at pH 5.0 yielded a colorless protein isolate (93.5% protein). The amino acid compositions of sunflower meal, concentrate, and isolate were similar. The phenol-free isolate and the untreated isolate exhibited identical minimum solubility points (pH 5.0) but the former showed a higher protein extractability above pH 7.0 than the latter. No significant differences appeared among electrophoretic patterns of albumin and globulin fractions from meal and protein concentrate.

Sunflower (*Helianthus annuus* L.) is the second largest oilseed crop as a world source of vegetable oil. Because of its high content of protein having good nutritional quality (Clandinin, 1958), sunflower meal also represents a protein source of great interest as a human food. However, the preparation of sunflower protein isolates for food products is prevented by the presence of undesirable phenolic compounds in the seed (Pomenta and Burns, 1971), such as chlorogenic and caffeic acids (Sechet-Sirat et al., 1959; Brummett and Burns, 1972). These acids bind to polar groups of the proteins at alkaline pH values usually employed for protein extraction, giving dark-green

or yellow products (Cater et al., 1970), thereby strongly reducing the available lysine content (Loomis and Battaile, 1966; Feeny, 1968).

Removal of these polyphenolic substances from sunflower meal has been attempted with organic solvents either in the presence or absence of reducing agents (Smith and Johnsen, 1948; Joubert, 1955; Gheyassudin et al., 1970; Girault et al., 1970). However these methods yield colored protein isolates, as color-forming phenols are only incompletely removed and cause denaturation of the proteins.

Colorless protein isolates were obtained from sunflower meals by Sosulski et al. (1972). These authors extracted the undesirable phenols by shaking whole kernels in 0.001 N HCl at different temperatures; however, partially denatured proteins were produced by this method (Kilara

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